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भारतीय मानक

सिंथैटिक इमल्शन रेजिन बाइंडर्स — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

SYNTHETIC EMULSION RESIN BINDERS — SPECIFICATION

(First Revision)

ICS 59.140.10

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Synthetic resin binders are used as film forming binders in various stages of finishing leathers, for example, bottom stock, pigment coat, top coat, etc, either alone or in admixture with protein binders. The synthetic resin binders are manufactured and marketed in various grades and qualities, yielding hard to soft films and used in varying formulations for finishing different types of leather. Realizing the wide application of this important auxiliary in the modern trend of leather finishing, the Committee felt it necessary to formulate this standard for the guidance of the leather finishing industry.

This standard was originally published in 1975. In this revision, the requirement on pentachloro phenol (PCP) has been introduced keeping in view of the demand for eco-friendly inputs from the leather industry.

The composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SYNTHETIC EMULSION RESIN BINDERS — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for synthetic emulsion resin binders for leather finishing.

1.1.1 This standard is not intended to cover resin emulsions which are used for tanning and impregnation.

2 REFERENCES

The standards listed below contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
101	Methods of sampling and test for paints, varnishes and related products:
(Part 1/Sec 1) : 1986	Test on liquid paint (general and physical), Sec 1 Sampling (*third revision*)
(Part 5/ Sec 2) :1988	Mechanical tests, Sec 2 Flexibility and adhesion (*third revision*)
323 : 2004	Rectified spirit for industrial use — Specification (*second revision*)
1640 : 2007	Glossary of terms relating to hides, skins and leather (*first revision*)
2631 : 2004	Specification for isopropyl alcohol (*first revision*)
14575 : 1999	Determination of pentacholorophenol (PCP) in leather — Method of test

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1640 and the following shall apply.

3.1 Resin Binder — Various synthetic polymer resins in emulsion state, prepared by emulsion polymerization of vinyl and/or other monomers.

4 REQUIREMENT

4.1 Physico-Chemical Characteristics

4.1.1 *Colour and Appearance*

The material when examined visually shall be opaque to translucent and free-flowing. It shall also be free from any sediments and foreign matter.

4.1.2 *p*H

The *p*H of the material when tested as prescribed in **A-1** shall be as agreed to between the purchaser and the supplier, but shall not vary by more than ±5 of the agreed value.

4.1.3 *Total Solids*

The manufacturer shall indicate the solid content which when tested in accordance with the test prescribed in **A-2** shall conform within a tolerance of ±1 percent.

4.1.4 *Residual Free Monomers*

The material shall be free from residual free monomers when tested in accordance with **A-3**.

4.1.5 *Stability to Specific Chemicals and Leather Finishing Formulations*

The material shall be considered satisfactory, if it passes the test prescribed in **A-4**.

4.1.6 *Freedom from Pentachlorophenol (PCP)*

The material shall not contain more than 5mg/kg of pentachlorophenol when tested in accordance with **A-5**.

4.1.7 *Storage Stability*

The material when kept in sealed original containers at 70 ± 2°C for 72 h, shall be stable without any breaking or coagulation of the emulsion (*see* Note).

NOTE — Such a material shall be stable at 27 ± 2°C when stored for a minimum period of 6 months from the date of manufacture.

4.1.8 *Freeze - Thaw Stability*

The material when tested in accordance with **A-6** shall pass the test.

4.1.9 *Cold Crack Resistance*

The material in the satisfactory concentration of finishing formulation shall not crack at -20°C when tested in accordance with **A-7**.

4.1.10 *Solvent Tolerance Test*

The material shall pass the test as prescribed in **A-8**.

4.2 Physical Characteristics of Resin Emulsion Film

The film of the material shall pass the test as prescribed in **A-9**.

4.3 Application Characteristics

The material in the satisfactory concentration of finishing formulation shall easily wet the surface without any froth or foam during brushing on to the leather surface.

5 PACKING AND MARKING

5.1 Packing

Unless otherwise specified the material shall be packed in containers as agreed to between the manufacturer and the purchaser.

5.2 Marking

Each container shall be marked with the following:

a) Name of the material:
b) Name of the manufacturer and/or trade-mark, if any;
c) Volume of the material;
d) Month and year of manufacture; and
e) Instructions for preparing the leather finishing formulation.

5.2.1 *BIS Certification Marking*

The containers may also be marked with Standard Mark.

5.2.1.1 The use of the Standard mark is governed by the provision of *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 101 (Part1/Sec 1).

ANNEX A

(*Clauses* 4.1.2 to 4.1.6 *and* 4.1.8 to 4.2)

TESTS FOR SYNTHETIC EMULSION RESIN BINDER AND ITS FILM

A-1 DETERMINATION OF *p*H

Determine the *p*H electrometrically, with the help of *p*H meter using glass electrodes.

A-2 DETERMINATION OF TOTAL SOLIDS

A-2.1 Outline of the Method

The filtered synthetic emulsion resin binder is heated in an aluminium dish at 100 ± 2°C for about 1½ h and weighed.

A-2.2 Procedure

Filter the synthetic emulsion resin binder through a funnel containing glass wool and collect the filtrate in a weighed aluminium dish. Collect about 2 g to 3 g of filtrate in the dish and weigh accurately. Heat the dish and contents in an oven maintained at 100 ± 2°C for about 90 min, till constant mass is obtained. Cool the dish to room temperature and weigh.

A-2.3 Calculation

$$\text{Total solids, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass of the residue, in g; and
M = mass of the sample taken for the test, in g.

A-3 DETERMINATION OF RESIDUAL FREE MONOMERS

A-3.1 Outline of the Method

The synthetic emulsion resin binder is diluted with an equal volume of water. Aqueous hydroquinone and anti-foaming agents are also added and the mixture is distilled. The distillate is collected in a graduated cylinder and examined for the presence of organic layer.

A-3.2 Apparatus

A-3.2.1 *Distillation Assembly,* modified Dean - Stark apparatus.

A-3.3 Reagents

A-3.3.1 *Aqueous Hydroquinone Solution*, 5 percent (*m/v*).

A-3.3.2 *Anti-Foaming Agent,* silicone type.

A-3.4 Procedure

Dilute 100 ml of the synthetic emulsion resin binder with equal amount of water. Add 2 ml of aqueous hydroquinone solution and 0.5 ml of anti-foaming agent. Take this in a distilling flask and distil the contents. Collect the distillate in a graduated cylinder, provided with recirculating provision. Recirculate the lower water layer to distilling flask and continue the distillation till the upper organic layer remains constant. This gives the percent monomer present in the emulsion. The synthetic emulsion resin binder shall be deemed to have passed the test, if no organic (upper) layer is present.

A-4 TEST FOR STABILITY TO SPECIFIC CHEMICALS AND LEATHER FINISHING FORMULATIONS

A-4.1 Outline of the Method

The synthetic emulsion resin binder is treated with the specific chemicals and leather finishing formulations and the emulsion is examined for any break or curdling.

A-4.2 Reagents

A-4.2.1 *Sodium Chloride Solution,* 5 percent (*m/v*).

A-4.2.2 *Calcium Chloride Solution,* 5 percent (*m/v*).

A-4.2.3 *Formic Acid Solution,* 5 percent (*m/v*).

A-4.2.4 *Sodium Carbonate Solution,* 5 percent (*m/v*).

A-4.2.5 *Ethyl Alcohol,* 5 percent (*m/v*).

A-4.2.6 *Isopropyl Alcohol,* 5 percent (*m/v*).

A-4.3 Procedure

Prepare the synthetic emulsion resin binder to contain 10 percent solid content. Take 3 ml for test with each of the above chemicals. To emulsion in a test tube add 10 ml of the test reagent, mix well and leave at 27 ± 2°C for 5 min. Examine the synthetic emulsion resin binder at the end of this period for stability. The synthetic emulsion resin binder shall be deemed to have passed the test in respect of the particular test reagent, if the synthetic emulsion resin binder shows no break or curdling.

A-5 DETERMINATION OF PENTACHLORO PHENOL (PCP)

A-5.1 Transfer about 5 g of the synthetic emulsion resin binder into a separating funnel of about 250 ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent *w/v* aqueous solution and ensure that the *p*H of the contents is acidic. Then carry out liquid-liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note). Transfer the aqueous layer into another separating funnel and carry out three more extractions of the aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps givn in **4.3.4.2** of IS 14575.

A-5.2 Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evaporator.

A-5.3 The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

A-6 TEST FOR FREEZE-THAW STABILITY

A-6.1 Outline of the Method

The synthetic emulsion resin binder is subjected to a cycle consisting of keeping at –10°C for 18 h and slowly bringing it to 25°C. Then the synthetic emulsion resin binder is examined for change in viscosity and coagulation.

A-6.2 Procedure

Dilute the synthetic emulsion resin binder to give 20 percent *(m/v)* solid content. Take this emulsion in a tin provided with a tight lid and coated inside with a chemical resistant lacquer. Replace the lid. Keep the tin in a thermostat at a temperature of –10 ± 2°C and keep at this temperature for 18 h. Then raise slowly the temperature to 25 ± 2°C. This constitutes one cycle. Examine the contents for change in viscosity, which should not show a change by more than 50 percent of the original, and for coagulation. Subject the synthetic emulsion resin binder to 3 such cycles and examine at the end for viscosity and coagulation.

A-6.2.1 The synthetic emulsion resin binder shall be deemed to have passed the test, if it has withstood 3 cycles satisfactorily.

A-7 TEST FOR COLD CRACK RESISTANCE

A-7.1 Outline of the Method

The synthetic emulsion resin binder in the satisfactory concentration of finishing formulations is subjected to sub-zero temperature, after application on test specimen and examined for any crack.

A-7.2 Apparatus

The testing apparatus consists of two parallel revolving cylinders, 25 mm in diameter and made of non-compressible material (glass, steel, hard PVC). The distance between the cylinders is variable.

A-7.3 Test Specimens

For preparing test specimen, cut pieces of size 15 × 150 mm from a sample of garment leather ready for finishing. Apply two coats of the finishing formulation prepared with the synthetic emulsion resin binder to be tested on the grain side, the second one being applied after the first coat dries up.

A-7.4 Conditioning

Condition the specimens at 27 ± 2°C and 65 percent relative humidity for 24 h before the test is carried out.

A-7.5 Procedure

Adjust the distance between the rollers to three times the thickness of the leather to be tested. Bend the leather with the finished side on the outer side, so that the two ends of the strip meet. Push the two ends through the space between the cylinders and fasten in a clamping device closely behind the cylinders. Thus, the leather strip forms a drop-shaped loop. Adjust the test chamber (refrigerator) to –20°C. Then, place the test apparatus with the leather test specimen in the test chamber and cool down to the test temperature for 30 min. Take it out and adjust the cylinders so that the leather loop is pulled through between the cylinders and then compressed in a space equal to the leather thickness. Take out the leather piece and check the test area for cracks in the finish coats. If the leather is resistant to cracking at the temperature tested, the test is repeated at a still lower temperature of 5°C. Repeat the test thrice with three test specimens. Report the results for each test temperature.

A-8 TEST FOR SOLVENT TOLERANCE

A-8.1 Outline of the Method

The synthetic emulsion resin binder is mixed with ethanol or isopropyl alcohol and examined for any deterioration like coagulation, thickening, etc.

A-8.2 Reagents

A-8.2.1 *Ethyl Alcohol,* conforming to IS 323.

A-8.2.2 *Isopropyl Alcohol,* conforming to IS 2631.

A-8.3 Procedure

Take 100 ml of the synthetic emulsion resin binder in a clean 250-ml beaker. Add 6 ml of ethyl alcohol with stirring. Ignore any initial local thickening. Examine the contents after a few minutes for coagulation, thickening, etc. Repeat the test using isopropyl alcohol also. The synthetic emulsion resin binder shall be deemed to have passed the test, if the test sample gives a smooth dispersion on dilution with ethyl alcohol and isopropyl alcohol.

A-9 TEST FOR PHYSICAL CHARACTERISTICS OF RESIN EMULSION FILM

A-9.1 Outline of the Method

A coat of the synthetic emulsion resin binder is applied on a tin-plated mild steel panel and the film formed is tested for flexibility and adhesion.

A-9.2 Procedure

A-9.2.1 Apply a coat of the synthetic emulsion resin binder on a tin-plated panel to give a film thickness of 50 to 75 microns and dry for 48 h. Bend the film over a mandrel of 6.25 mm diameter as prescribed in **16** of IS 101 (Part 5/Sec 2). Examine the film and report failure of the test, if there be any sign of visible damage or detachment of film.

A-9.2.2 Pour some quantity of well mixed synthetic emulsion resin binder in a cavity of size 12.0 × 5.0 × 0.1 cm made of glass plates. Place the cavity in a horizontal position and dry at 27 ± 2°C. Peel off the film from the plate for testing.

A-9.2.3 The synthetic emulsion resin binder shall be deemed to have passed the test, if it does not crack or detach when tested as in **A-9.2.1**, and when tested as in **A-9.2.2**, the film is transparent, glossy and free from haziness.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

Organization	*Representatives(s)*
Central Leather Research Institute, Chennai	Director (***Chairman***)
A. V. Thomas Leather & Allied Products Pvt Ltd, Chennai	Shri Habib Hussain Shri K. Manivannan (*Alternate*)
All India Skins & Hide Tanners and Merchants Assciation, Chennai	Shri Mohan M. Sreenivas Shri S. Mohammed Hassan (*Alternate*)
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Leather Chemicals Manufacturers Association, Mumbai	Shri Vaduvur T. Srikanth Shri Sanjeev Mehta (*Alternate*)
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Organization	*Representatives(s)*
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